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**HYDROLYTIC STABILITY OF ADIPRENE L-100
AND ALTERNATE MATERIALS**

PDO 6989249, Final Report

D. J. Caruthers, Project Leader

Project Team:
G. W. Griffith
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R. M. Smith

Published August 1976

DEPARTMENT OF DEFENSE
ELASTICS TECHNICAL EVALUATION CENTER
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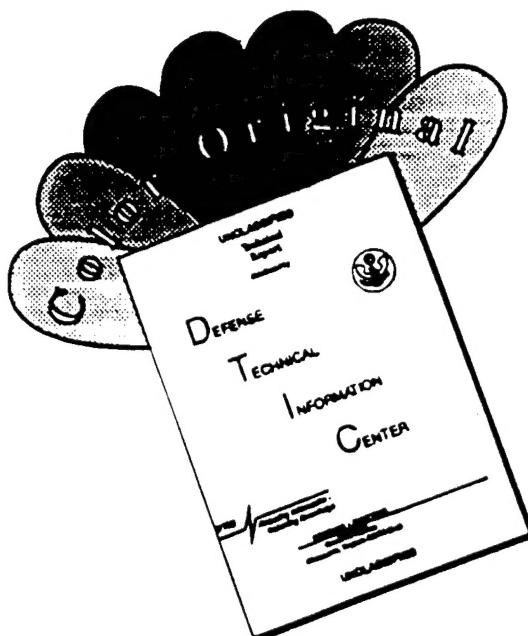
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Final Report

Technical Communications



**Kansas City
Division**

HYDROLYTIC STABILITY OF ADIPRENE L-100 AND ALTERNATE MATERIALS
BDX-613-1489, UNCLASSIFIED Final Report, Published August 1976
Prepared by D. J. Caruthers, D/814, under PDO 6989249

Adiprene L-100/MOCA (E. I. du Pont de Nemours and Company) and four potential alternate materials were tested after accelerated aging conditions in a steam autoclave. Test methods included: mechanical spectrometry, thermomechanical analysis, infrared analysis, solubility in tetrahydrofuran, and gel permeation chromatography. Conathane EN-7 was the most resistant to accelerated attack by steam hydrolysis and showed little change in properties after 50 hours exposure.

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SUMMARY

In order to more thoroughly evaluate alternate materials for du Pont's Adiprene L-100/MOCA, resistance to accelerated aging was measured using dynamic modulus tests. The change in modulus was compared to changes in the basic polymer.

Previous work had only reported changes in hardness. This report describes structural changes that occurred. The changes were tested using thermomechanical analysis, infrared spectrometry, and by determining the solubility of the polymer. Also, the molecular size distribution of soluble materials determined by gel permeation chromatography is presented.

All of the elastomers were subjected to steam in an autoclave at 121°C at 15 psig (207 kPa) for up to 216 hours. It was found that Uralite 3121S, Adiprene L-315/polyol, and Adiprene L-100/MOCA had a low degree of hydrolytic stability. Adiprene LW-520/MDA was moderately stable and Conathane EN-7 was the most stable of the materials tested.

DISCUSSION

SCOPE AND PURPOSE

Urethane elastomers degrade when exposed to high humidity and temperature. This project was undertaken to measure the degradation under conditions of accelerated exposure using 121°C steam. Adiprene L-100/MOCA was compared with two urethane encapsulants, Uralite 3121S and Conathane EN-7, as well as with two urethane adhesives, a polyol cured Adiprene LW-520 and L-315 cured with trimethylolpropane (TMP).

PRIOR WORK

Hydrolytic stability had been studied by comparing changes in hardness at Sandia, Albuquerque, however, no data was available to determine what changes occurred to the modulus and chemical structure of the four materials.

ACTIVITY

Materials

Four materials recommended as alternates for the Adiprene/MOCA system were prepared in order to determine their hydrolytic stability using an accelerated aging environment of 15 psig (207 kPa) steam. The systems selected were based upon a degree of successful production and encompass two used as casting compounds and two recommended for use as room temperature curing adhesives (Table 1).

Materials were prepared by mixing the hardener with the resin component under vacuum conditions to eliminate inclusion of air into the polymer mixtures and by injecting into pre-evacuated molds. Test specimens were cured in an 8-cavity bar mold. Each bar was approximately 0.437 inches wide by 0.125 inch thick by 8 inches long (11.1 by 3.2 by 203 mm) (Figure 1).

Torsional Shear Modulus

Shear modulus specimens were cut into 1.5 inch (38.1 mm) lengths for testing. For each modulus test, either as-cast or after aging, a new specimen was taken to avoid error caused by grip marks left by any previous test. One specimen of each material and cure was tested on the mechanical spectrometer over a temperature range of -140° to 140°C at a frequency of 0.1 hertz using the fixed torsional pendulum mode. This data was recorded as the as-cured data for each material. Separate specimens were placed in individual aluminum dishes and aged in an autoclave at

Table 1. Curing Time and Temperature of Specimens
Used for Steam Hydrolysis

Specimen (Figure 2)	Cure Schedule		Steam Hydrolysis (Hours)
	(Hours)	(°C)	
A. Uralite 3121S*	+++	RT	7
B. Conathane EN-7**	16	80	24
C. PR-1660***	3	100	24
D. Uralite 3121S	3		7
E. Vibrathane B-635/3080†	+++	RT	24
F. Adiprene LW-520/ LD-2729††	16	100	24
G. Adiprene L-100/ MOCA	4	71	24
H. Adiprene L-315/TMP, 1,4-BDO, FeAcAc	6	100	24
I. Conathane EN-8	+++	RT	8

Elevated temperature cures were used for L-315 and LW-520 to force these materials to stabilize within a practical time schedule. PR-1660, Vibrathane B-635/3080, and Conathane EN-8 were not analyzed.

*Hexcel Corporation, Rezolin Division

**Conap Incorporated

***Products Research Company

†Uniroyal Incorporated, Uniroyal Chemical

††E. I. du Pont de Nemours and Company Incorporated

+++One week minimum

121°C at 15 psig (207 kPa) in steam. Specimens were removed at the time intervals specified and tested for changes in shear modulus (Figure 2) (Table 1).

Visual effects occurred as small blisters in the room temperature cured Uralite 3121S and to a lesser extent in the Uralite 3121S cured at elevated temperature. Adiprene L-100/MOCA, Conathane EN-7, and Adiprene L-315 showed no visual effects; however, the amine cured Adiprene LW-520 showed gross shrinkage so another specimen, subjected to only 7 hours steam, was used to measure the change in shear modulus (Figures 3 through 8).

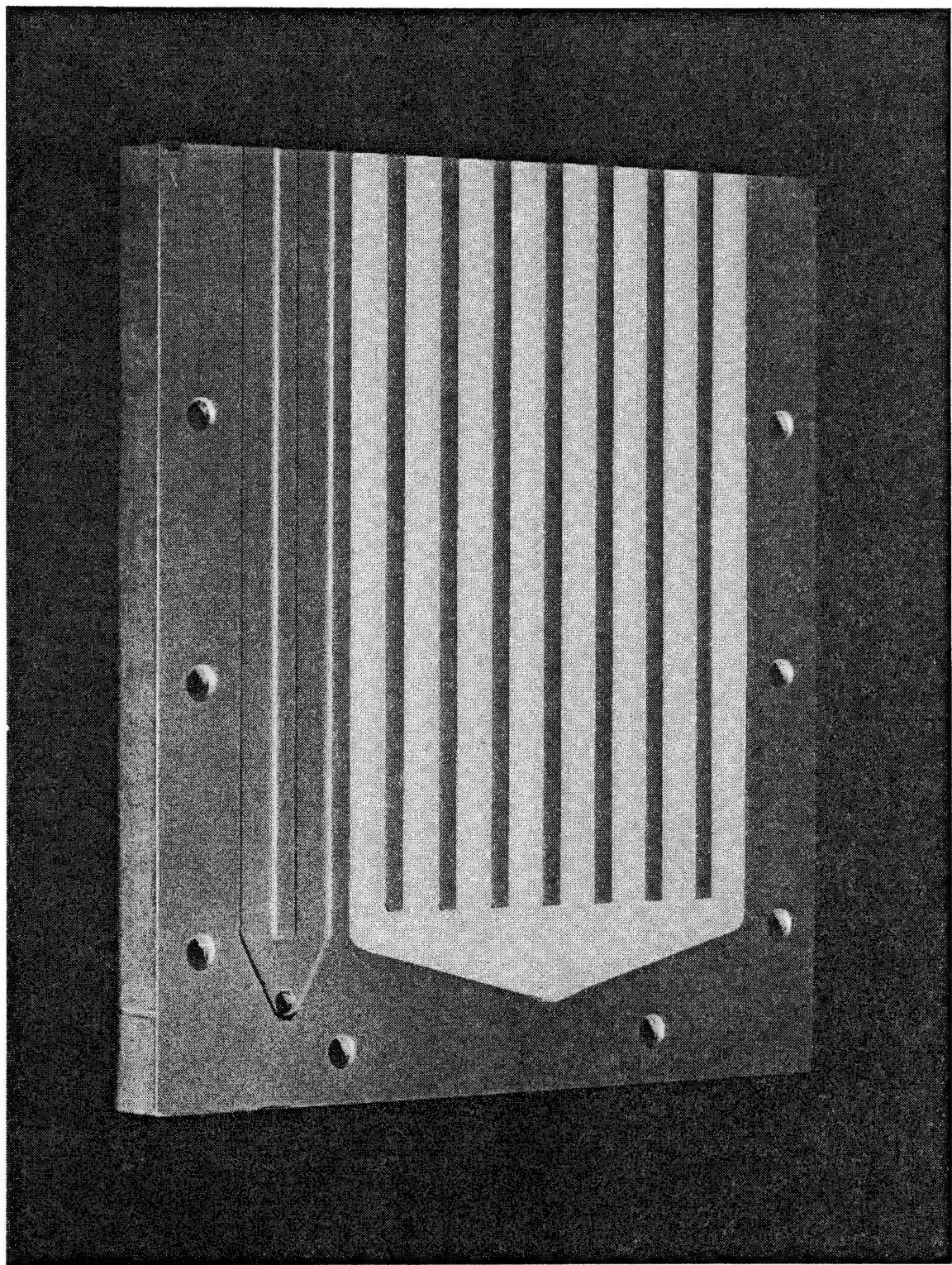


Figure 1. Mold for Casting Shear Modulus Test Bars

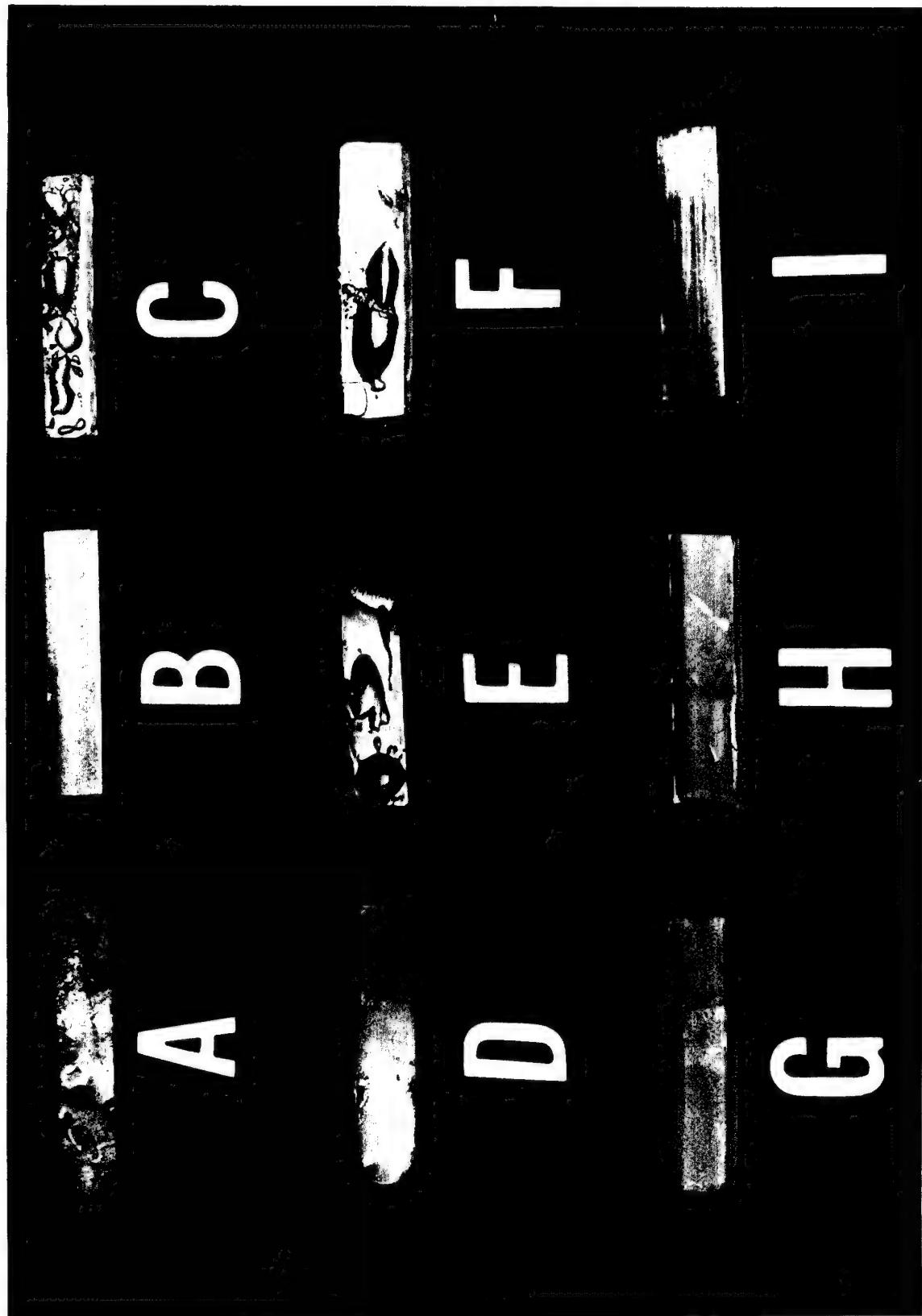


Figure 2. Effects of Exposure to Steam Hydrolysis

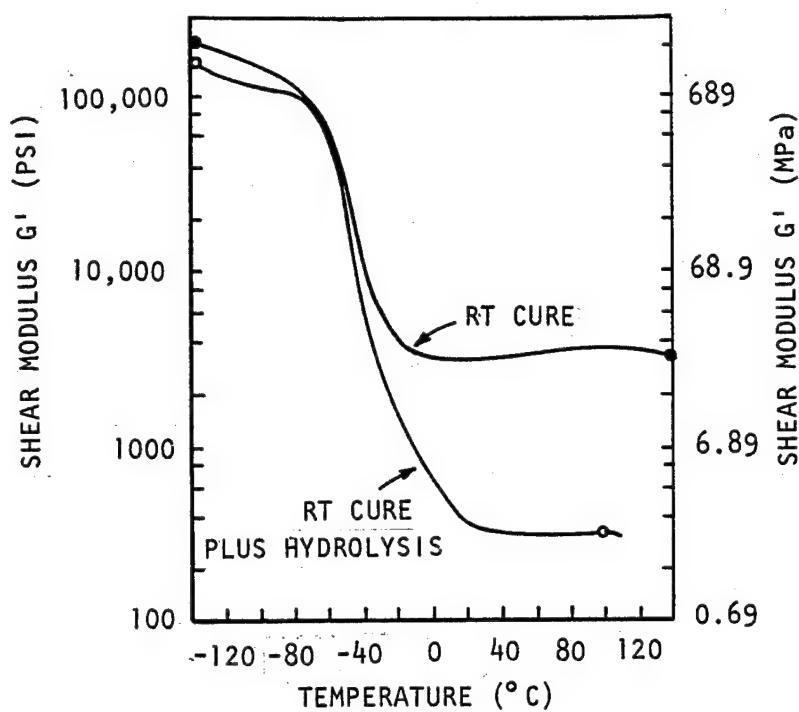


Figure 3. Torsional Modulus Versus Temperature,
Adiprene L-100/MOCA

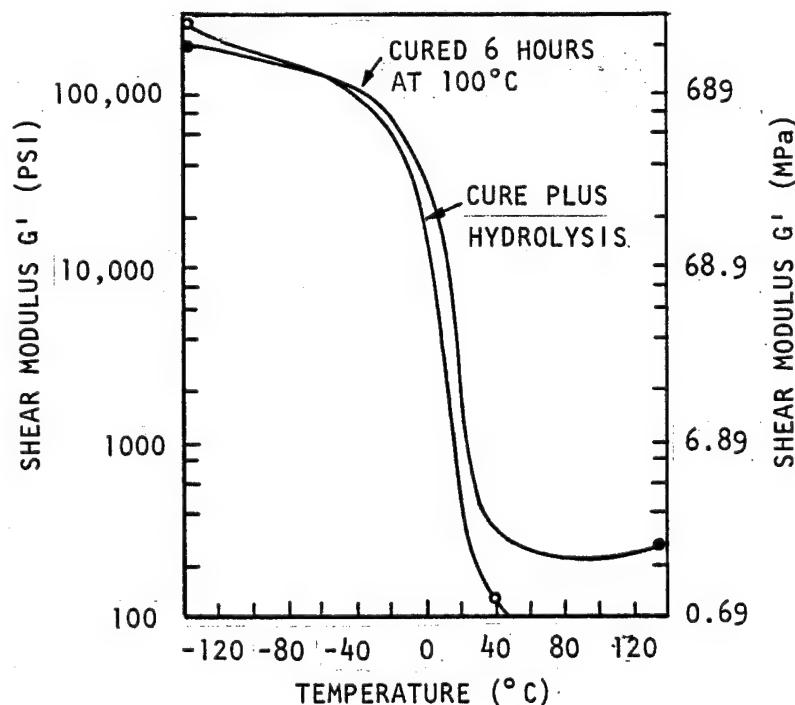


Figure 4. Torsional Modulus Versus Temperature,
Adiprene L-315/TMP, 1,4-BDO, FeAcAc

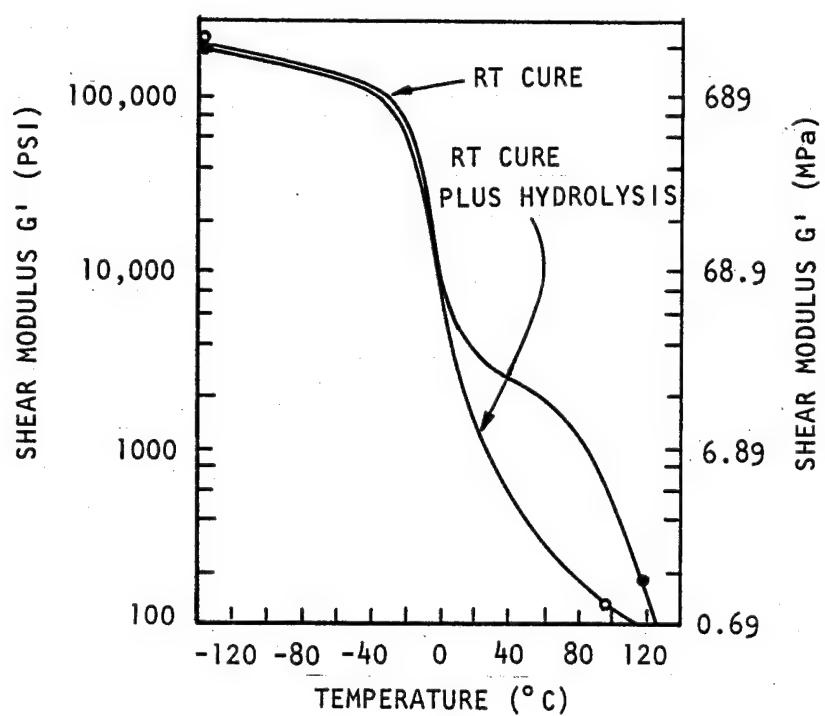


Figure 5. Torsional Modulus Versus Temperature,
Uralite 3121S Room Temperature Cure

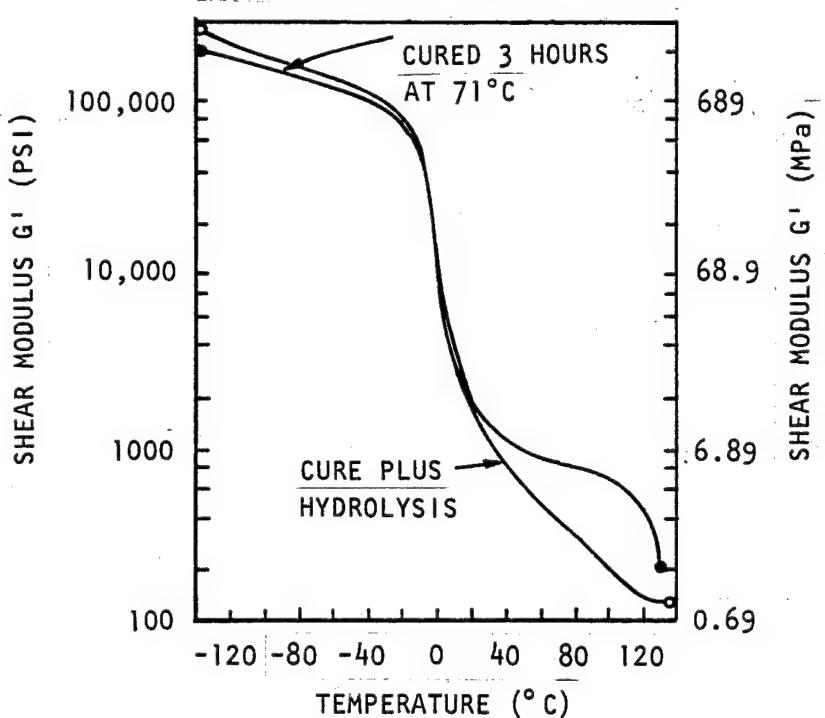


Figure 6. Torsional Modulus Versus Temperature,
Uralite 3121S 71°C Cure

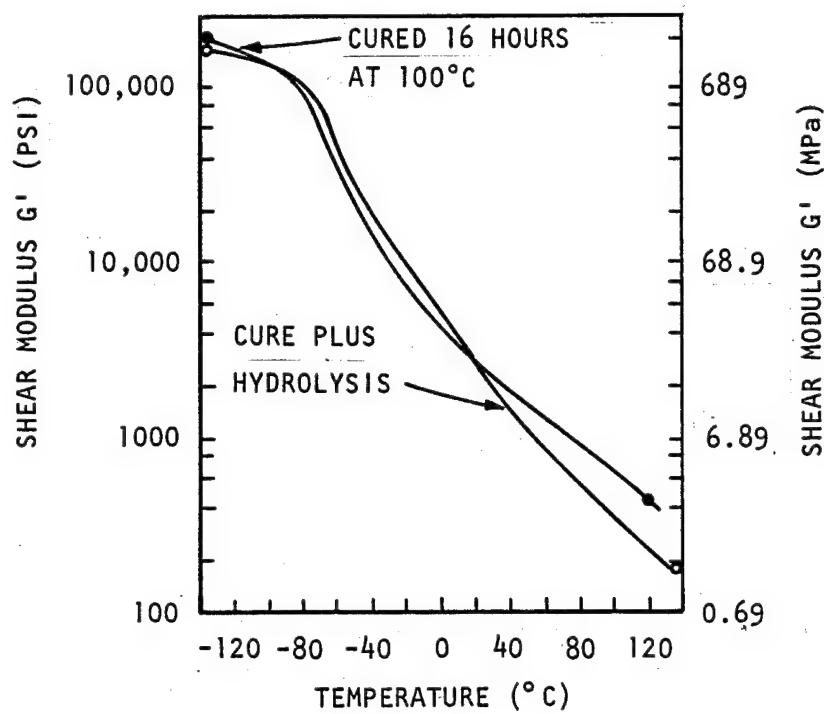


Figure 7. Torsional Modulus Versus Temperature,
Adiprene LW-520/LD-2729

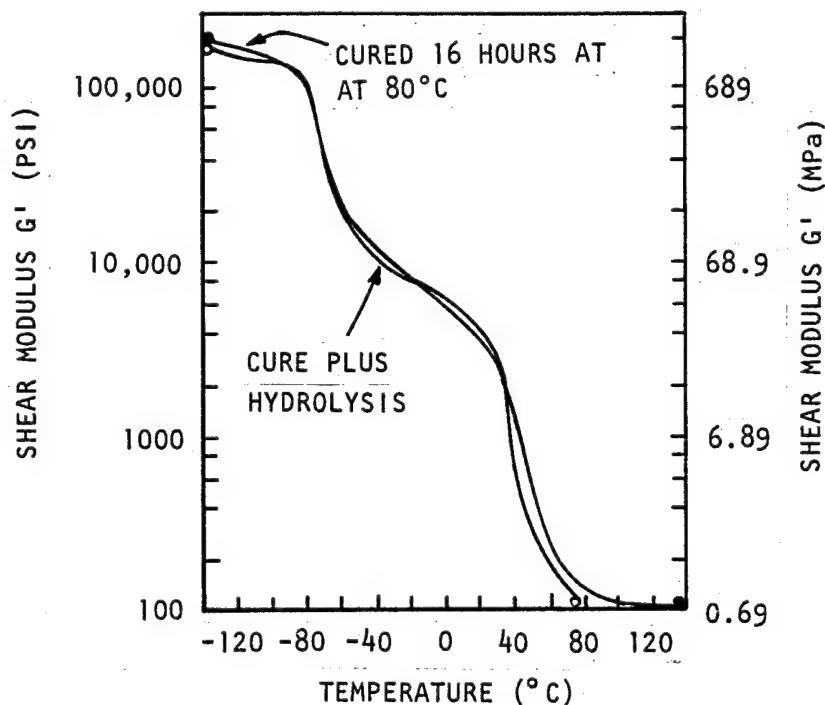


Figure 8. Torsional Modulus Versus Temperature,
Conathane EN-7

The steam environment caused the largest modulus change to Adiprene L-100/MOCA (Figure 3) dropping the shear modulus one order of magnitude from 3000 to 300 psi (20.6 to 2.06 MPa). The values for as-cast Adiprene L-100/MOCA leveled at 0°C and remained level or increased slightly as the temperature was raised to 140°C. After being subjected to 24 hours of 15 psig (207 kPa) steam the shear modulus of the material leveled at 20°C and remained level through 100°C. At 120°C the modulus dropped below the recording range of the rheometer.

Conathane EN-7 showed little change after a 24 hour steam exposure from as-cast data (Figure 8); therefore, it is considered to be the most stable of the materials tested. It is noted that, although degradation of the Adiprene L-100/MOCA exists, the modulus still levels at 300 psi (2.06 MPa) while the modulus of Conathane EN-7 drops below 100 psi (0.69 MPa) above 80°C. Although little change in modulus occurs due to this accelerated exposure, care must be exercised when selecting the EN-7 as an encapsulant for use at elevated temperature.

Uralite 3121S (Figure 5) is a room temperature curing urethane resin system showing a second order transition temperature near 0°C; this is higher than either Adiprene L-100 or Conathane EN-7. It was noted that after 24 hours steam exposure, severe blistering occurred and some evidence of blistering was evident after only 7 hours as shown in Specimen A, Figure 2.

Uralite 3121S (Figure 6) which was cured at 71°C for 3 hours showed a slightly different torsional modulus between 0° and 100°C. The shear modulus values separate near 0°C, the room temperature cured material maintaining almost twice the value of 71°C cured material. The changes caused by 7 hours steam exposure were similar for both cures which indicated there was no distinguishing difference between cures after hydrolysis.

The catalyzed diol cured Adiprene L-315 (Figure 4) indicates a second order transition between 0° and 20°C which is slightly above any other material tested. This formulation when cured for 6 hours at 100°C shows the characteristic flat modulus of Adiprene L-100/MOCA above 40°C but one order of magnitude lower in value between 200 and 300 psi (1379 to 2067 kPa). Material subjected to 24 hours of steam aging followed the same shear modulus curve from -140° through 0°C as unaged material. The shear modulus of aged material drops off below 100 psi (689 kPa) above 40°C. The mechanical rheometer as adjusted had a practicable lower limit of 100 psi (689 kPa).

Adiprene LW-520 cured with MDA (Figure 7) indicated considerable damage from the 24 hour steam environment as shown by Specimen F in Figure 2. An additional specimen was subjected to 7 hours of steam and a plot of modulus shows a general deterioration of the material above 20°C increasing in effect through 140°C which was the highest temperature tested. Adiprene LW-520 does not have a level section in the shear modulus curve, at the temperatures tested, as do Adiprene L-100 and L-315. This may be because LW-520 uses an aliphatic instead of aromatic diisocyanate as used in the other Adiprene materials tested.

Analysis

Additional 1.5 by 0.437 by 0.125 inch (38.1 by 11.2 by 3.18 mm) specimens were used to study the degradation of the elastomers. These were aged in an autoclave at 121°C and 15 psig (207 kPa) steam, samples being taken at the recorded intervals. Samples removed from the autoclave were dried at 80°C and monitored by thermomechanical analysis, infrared spectrum change, solubility and tackiness, and molecular size distribution of soluble materials. Tests were continued until a major change was detected or until the specimen had been subjected to 216 hours of the steam environment.

The primary analytical method was by thermomechanical analysis (TMA) (Figures 9 through 14). In this test a small sample of the material was cut smooth and with relatively parallel faces. A quartz probe of 0.025 inch diameter (635 μm) was lowered onto the sample and loaded with a 1 gram mass, which gave a tip pressure of 14 psi (96.5 kPa). The sample was cooled to -100°C and then heated at a rate of 10°C per minute, until the softening point had been recorded. The recorded trace indicated dynamic changes in the vertical position of the probe as the sample swelled or softened beneath it. Usually a trace will show expansion at low temperature followed occasionally by a dip in the curve between -25° and 25°C, which can be attributed to a glass transition phase change. Somewhere in the elevated temperature range (usually over 100°C) the elastomer will undergo softening, and the trace will show a sharp downward trend as the probe penetrates the sample. The shape and temperature of this softening point is a good indication of a change in physical properties, when compared to a standard curve of the same material that has not seen any degradation (hydrolysis in this case). This method worked very well and was quite sensitive with most of the samples tested.

Infrared spectra change were also used to study the hydrolytic cleavage in the elastomers (Figures 15 through 19). Since the untreated specimens were not soluble in any solvent due to cross-linking, it was decided to microtome some very thin slices of the elastomers. Typically, slices from 10 to 30 microns (μm) were

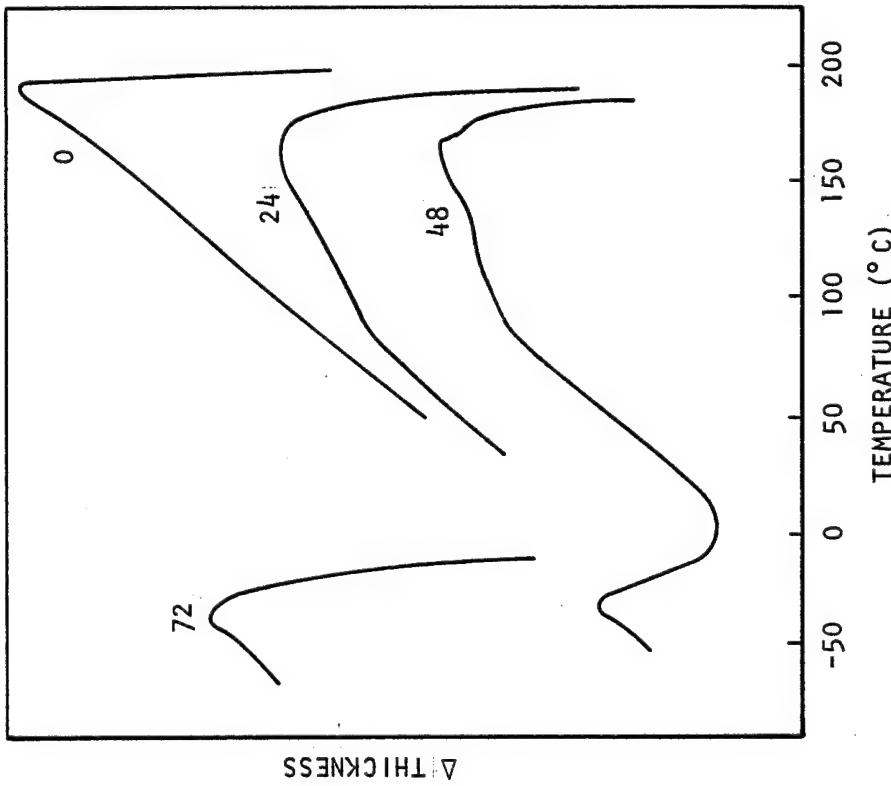
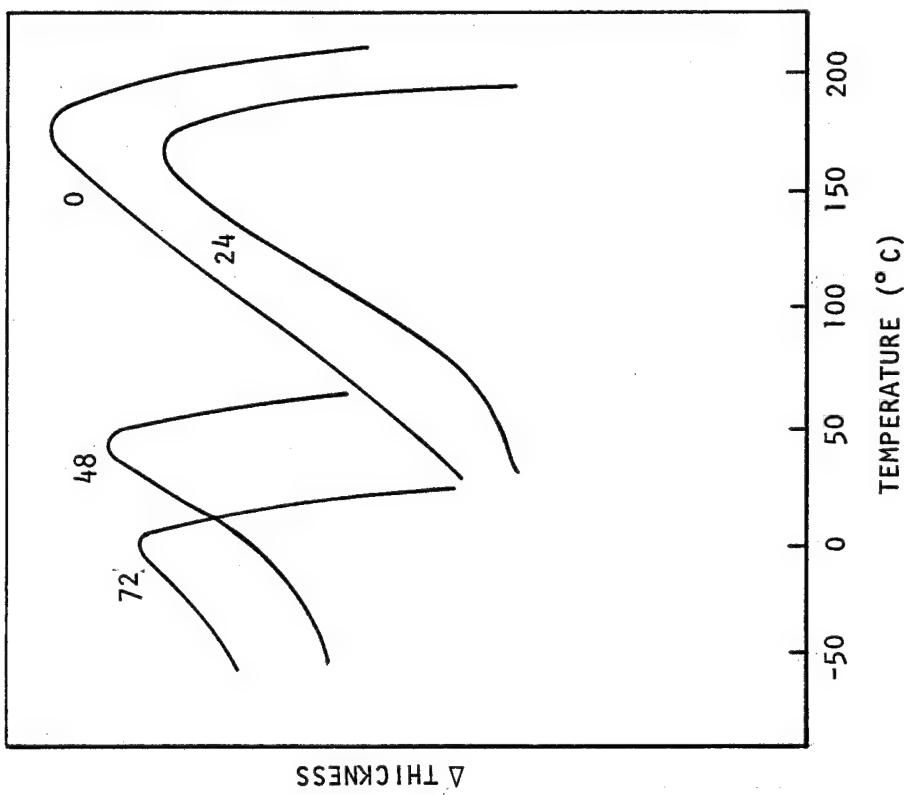


Figure 9. TMA of Cured Adiprene L-100/MOCA After Steam Hydrolysis at Indicated Times (Hours)

Figure 10. TMA of Room Temperature Cured Adiprene L-315/TMP, 1,4-BDO, FeAcAc After Steam Hydrolysis at Indicated Times (Hours)

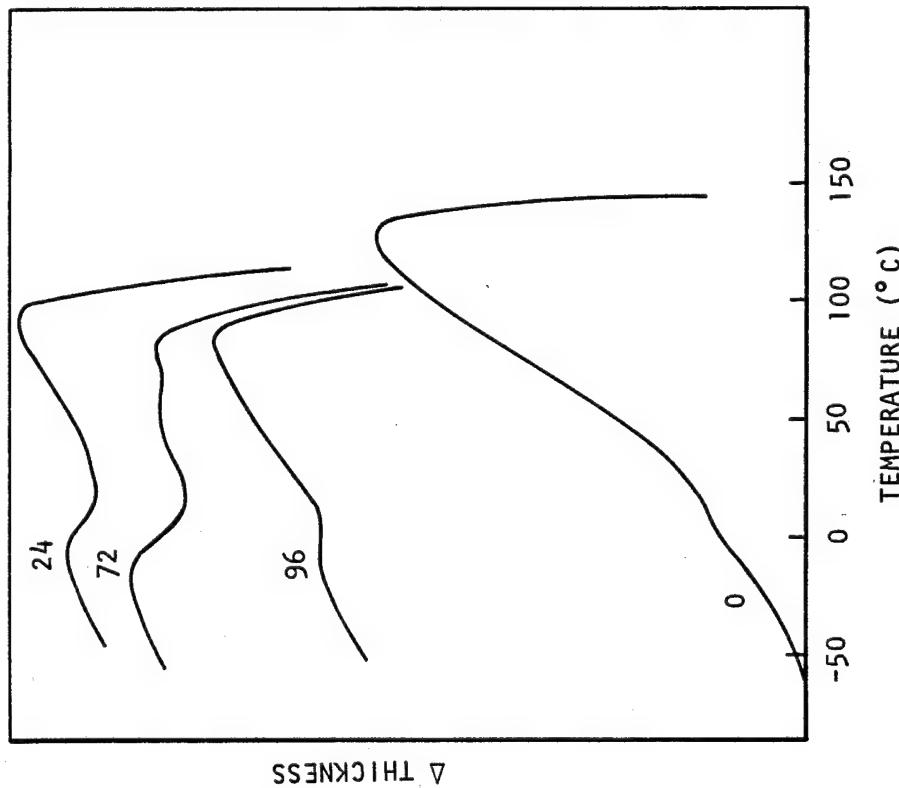
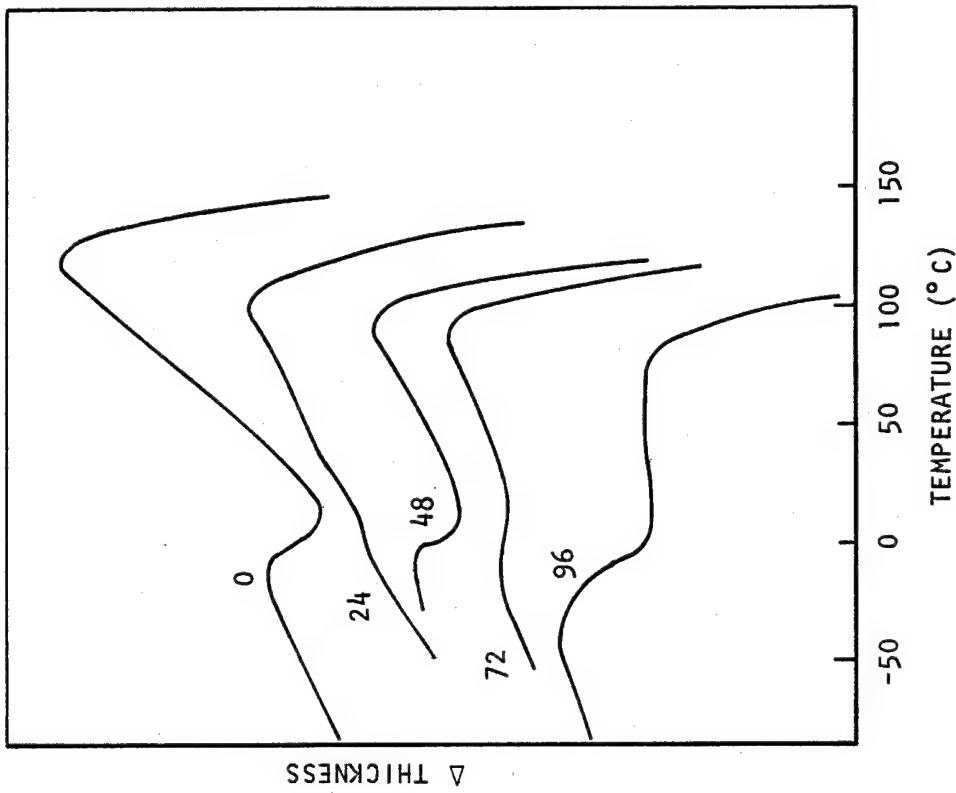


Figure 11. TMA of Room Temperature Cured Uralite 3121S After Steam Hydrolysis at Indicated Times (Hours)

Figure 12. TMA of 71°C Cured Uralite 3121S After Steam Hydrolysis at Indicated Times (Hours)

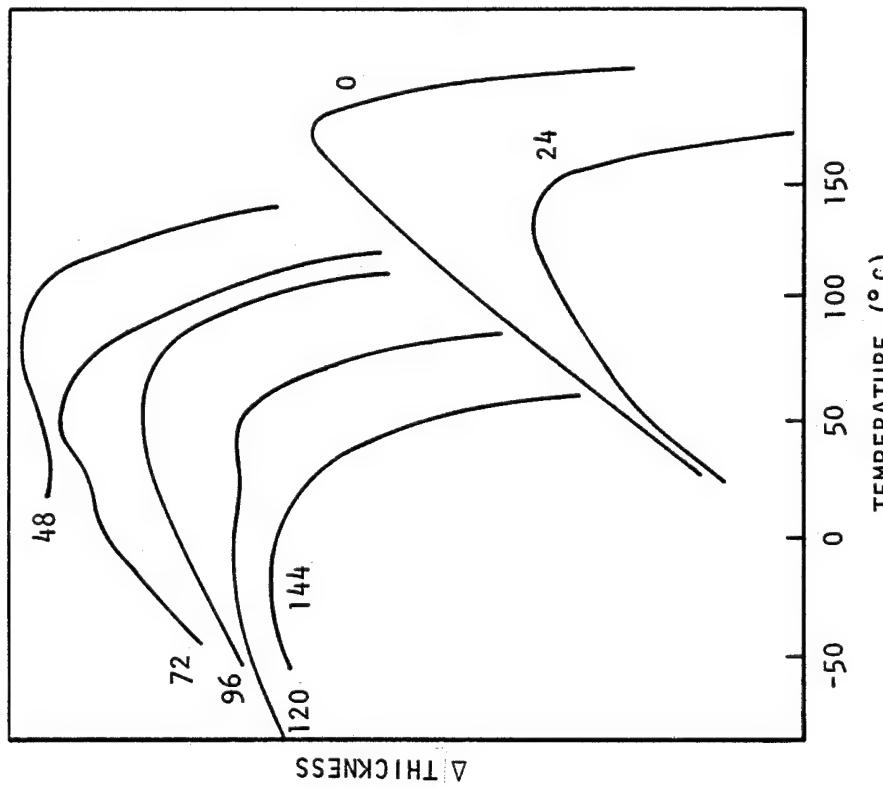


Figure 13. TMA of Cured Adiprene LW-520 After Steam Hydrolysis at Indicated Times (Hours)

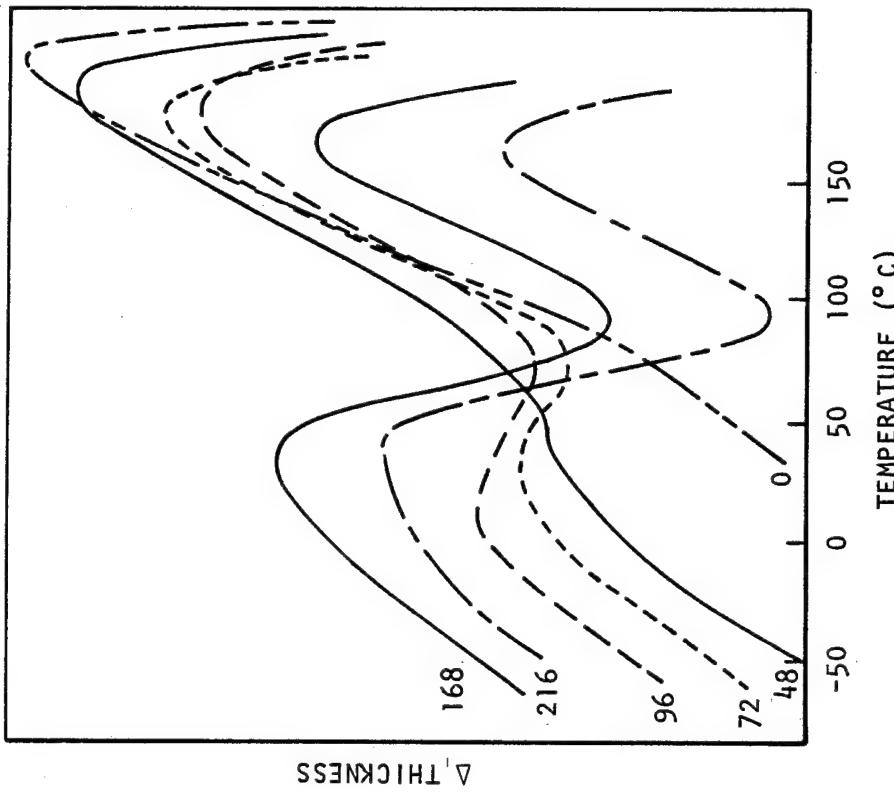


Figure 14. TMA Analysis of Cured Conathane EN-7 After Steam Hydrolysis at Indicated Times (Hours)

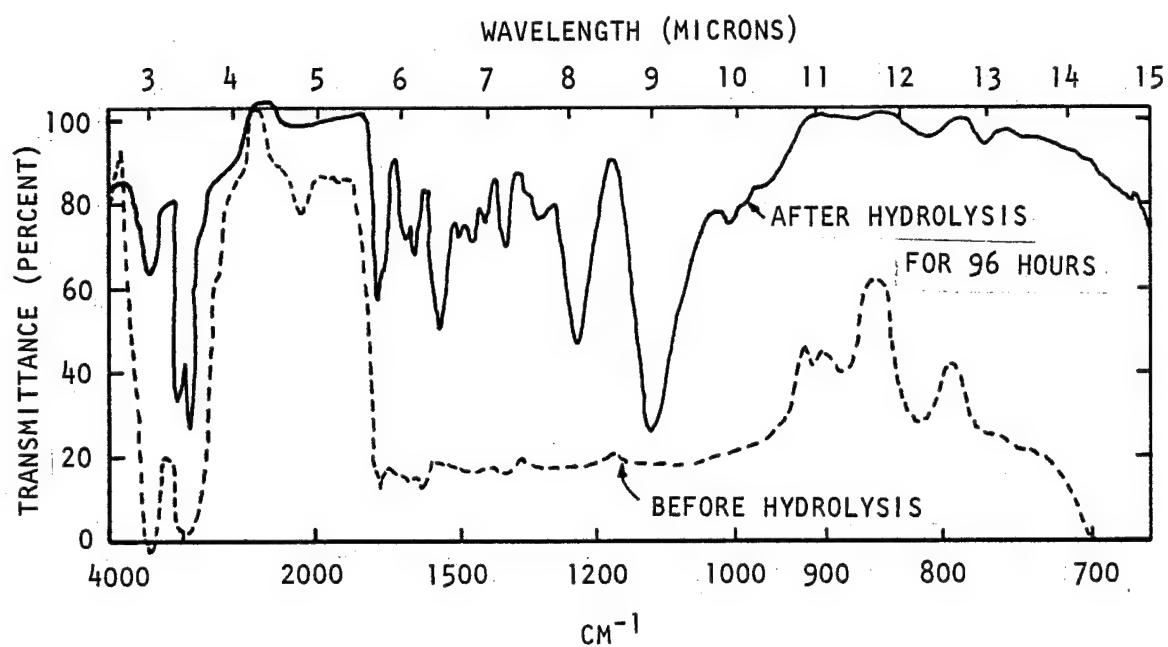


Figure 15. Infrared Spectra, Adiprene L-100/MOCA

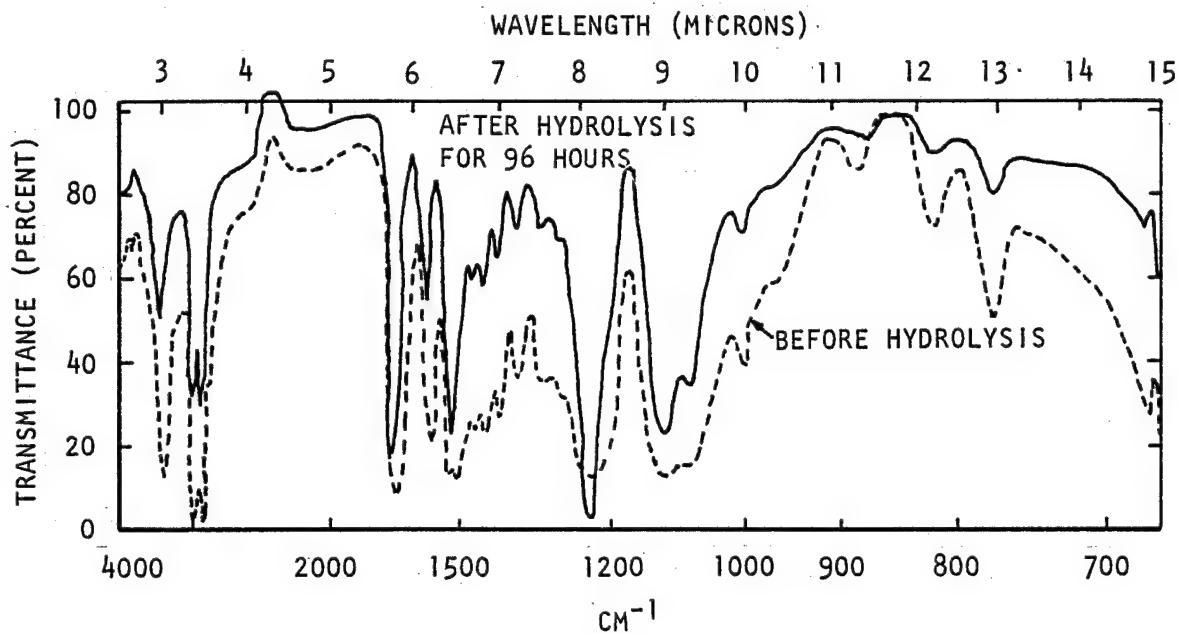


Figure 16. Infrared Spectra, Adiprene L-315/TMP, 1,4-BDO, FeAcAc

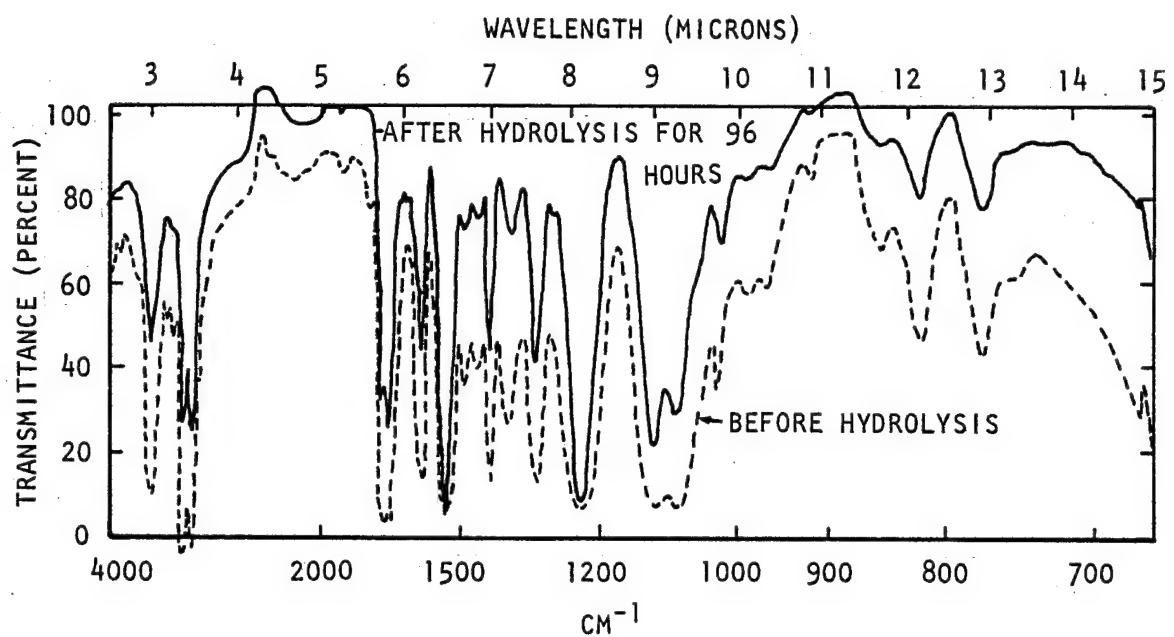


Figure 17. Infrared Spectra, Uralite 3121S

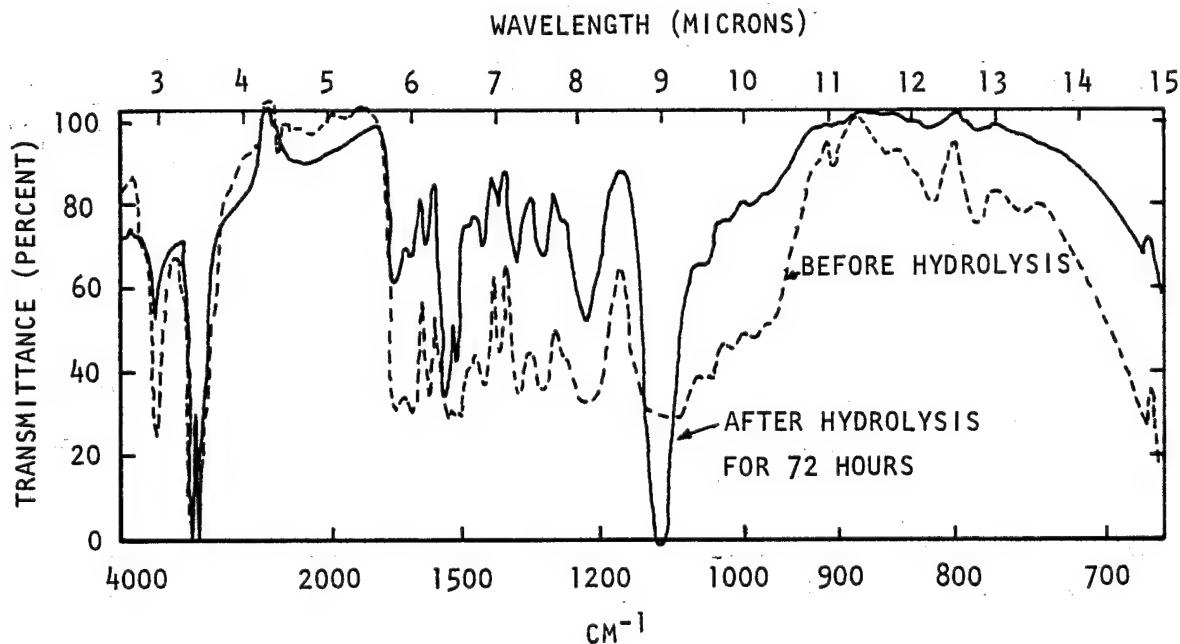


Figure 18. Infrared Spectra, Adiprene LW-520/LD-2729

necessary for good quality spectra. The samples were scanned from 2.5 to 15 microns and compared with spectra of the hydrolyzed samples. In some cases the hydrolyzed samples were very soft and soluble, so smears were made rather than microtome slices. The infrared method was useful only to show major changes in the properties of the samples and was not as sensitive or as useful as the TMA method.

Another indication of hydrolysis was the solubility of the polymer and its physical tackiness. Once the cross-links in the elastomer have been cleaved, it becomes readily soluble in solvents such as tetrahydrofuran (THF) or dichloromethane. When the backbone of the elastomer cleaves, the material will become soft and waxy, followed by surface tackiness, and finally complete liquefaction of the elastomer. These tests are a good indication of how severely the elastomer has been ruptured by the hydrolysis. Complete hydrolysis would give polyols and amines as final products.

The last indication of hydrolysis used was the molecular size distribution of THF soluble materials, as determined by gel permeation chromatography (GPC) (Figures 20 through 23). Since GPC chromatograms could not be obtained for the insoluble untreated elastomers, the curves could only be compared to curves of the prepolymer starting materials. Presumably if the initial elastomers could be run, then their peaks would probably occur at elution count 23 or lower. Occasionally small peaks were evident at counts 37 or 38, which were due to cure agents, indicating cleavage of the backbone of the elastomer molecule.

Characteristics of Materials

Adiprene L-100/MOCA

This formulation consists of a polyoxybutylene glycol capped with 2,4-toluene-diisocyanate (TDI), which is then extended with 4,4'-methylene bis (orthochloroaniline), better known as MOCA. It is cured at 71°C for 4 hours. The TMA trace (Figure 9) displayed a sharp softening point at 190°C. After 24 hours of hydrolysis, the sample became slightly yellow and more rubbery. The softening point was much broader and peaked at 74°C which was a considerable lowering. At 48 hours the sample became soft, waxy, and soluble in THF, indicating considerable breakdown of the molecule. The softening point was broader and slightly lower. It also exhibited a depression at -35°C, which was probably the glass transition point. Finally, at 72 hours the material became very sticky and soluble. The softening point decreased to -35°C. GPC traces (Figure 20) indicated the material was very low in molecular weight, nearly as low as the prepolymer. MOCA was also seen in the trace, which meant that the molecule cleaved at the bond between the nitrogen of the MOCA and the carbonyl of the TDI.

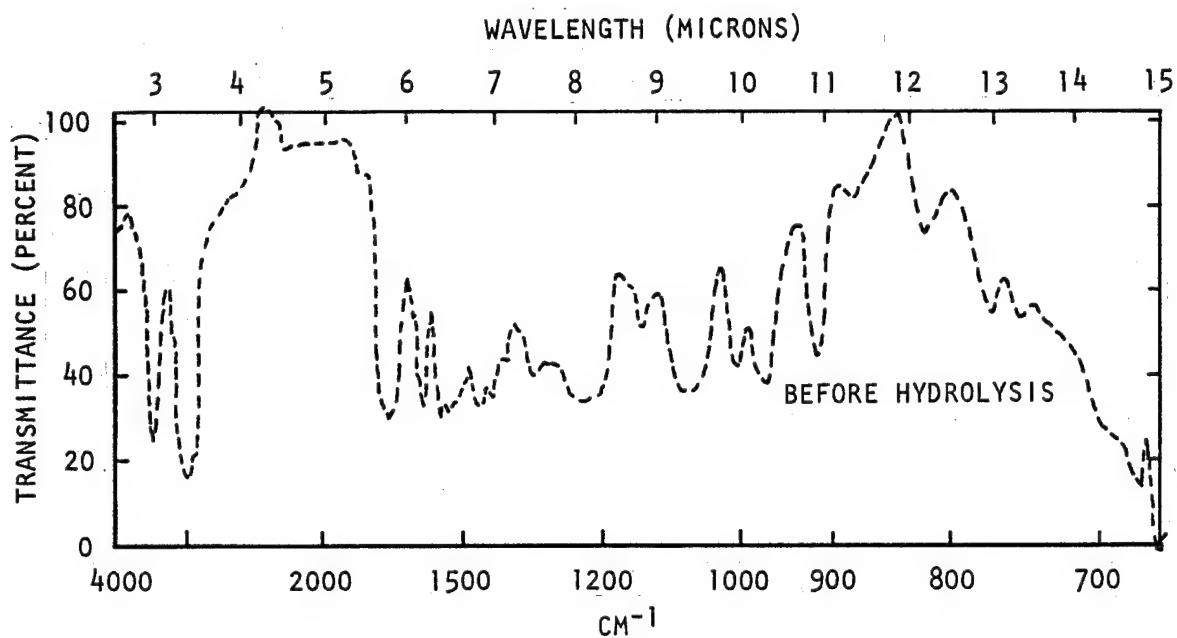


Figure 19. Infrared Spectra, Conathane EN-7

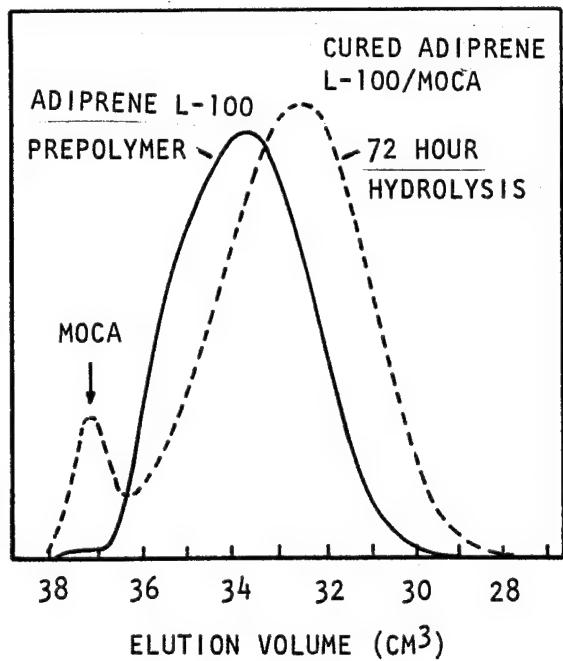


Figure 20. GPC Analysis of Elution Volume Changes Caused by Steam Hydrolysis of Adiprene L-100

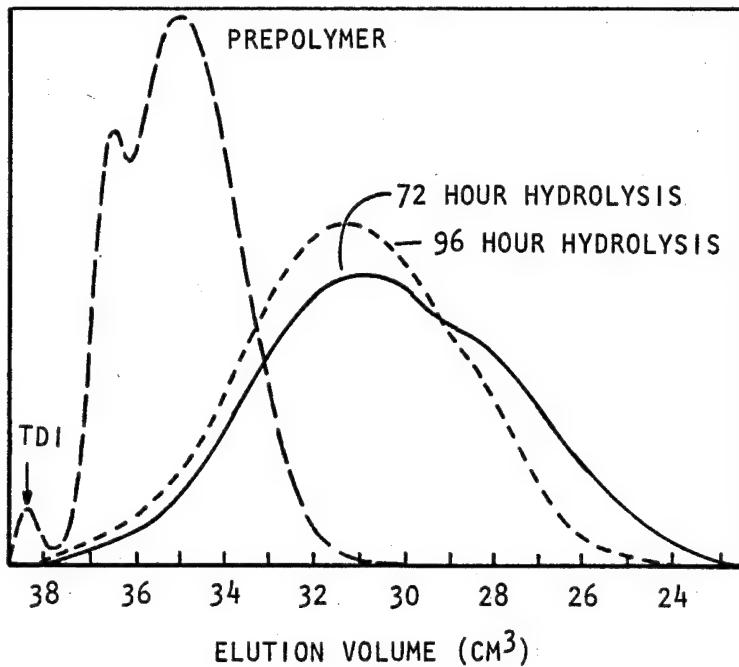


Figure 21. GPC Analysis of Elution Volume Changes Caused by Steam Hydrolysis of Adiprene L-315

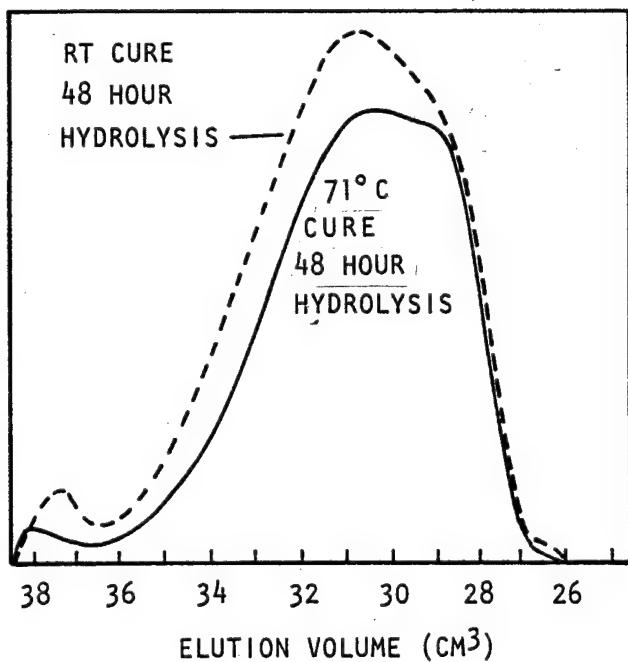


Figure 22. GPC Analysis of Elution Volume Changes Caused by Steam Hydrolysis of Uralite 3121S

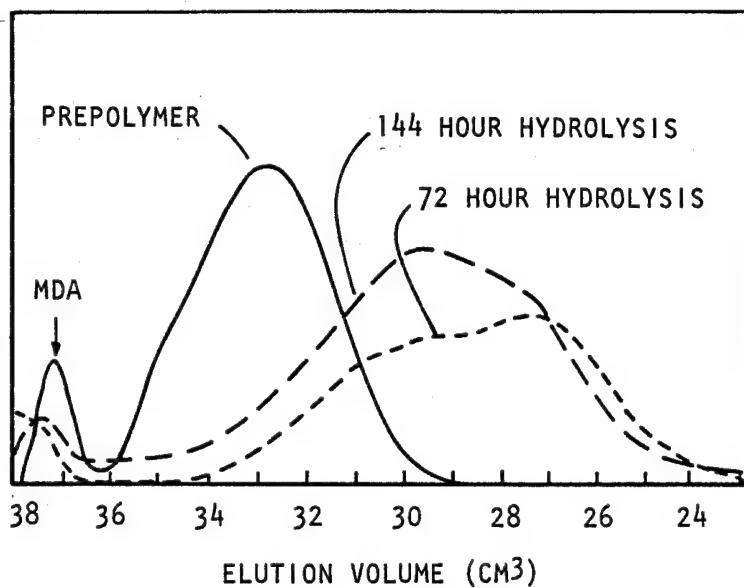


Figure 23. GPC Analysis of Elution Volume Changes Caused by Steam Hydrolysis of Adiprene LW-520

Since no diaminotoluene was seen, the bond between the TDI and the polyoxybutylene remained intact. The infrared spectra (Figure 15) indicated a low molecular weight urethane, similar to a prepolymer, for a 96 hour hydrolyzed specimen.

Adiprene L-315/Polyol, FeAcAc

The L-315 elastomer consists of a TDI capped polyoxybutylene glycol, which is extended with a mixture of 70 percent 1,4-butanediol and 30 percent trimethylpropane (TMP). The system also contains iron acetylacetone (FeAcAc) curing agent and is cured at 100°C for 6 hours. The unhydrolyzed sample had a softening point which peaked at 180°C (Figure 10). The 24 hour hydrolyzed sample exhibited a slightly lower softening point of 167°C, which was nearly as sharp. The sample was also slightly less resilient. After 48 hours the sample became quite soluble and had a softening point at 45°C, indicating that the material had suddenly depolymerized. At 72 hours the softening point had shifted to 0°C, and the sample had melted and become slightly sticky. GPC traces (Figure 21) were obtained for this sample as well as a 96 hour sample. At 72 hours the chromatogram indicated a significant amount of high molecular weight fragments, which virtually disappeared after 96 hours of hydrolysis. The principle peak was not as low as the prepolymer peak shown, but further hydrolysis should close the gap. Neither GPC or infrared (Figure 16) indicated the presence of butanediol, TMP, or diaminotoluene;

therefore, complete cleavage of the extender polyols had not occurred, unlike the case for MOCA cured Adiprene L-100. This means that the oxygen to carbonyl bond is not as easily split as the nitrogen to carbonyl bond.

Hexcel 3121S

Hexcel 3121S is a 4,4'-diphenylmethane diisocyanate (MDI) capped polyoxybutylene glycol which is extended with butanediol. The system is cured at room temperature or for 3 hours at 71°C. Actually the diisocyanate is not pure MDI but a modified MDI, which is a mixture of MDI and a trifunctional cycloadduct of MDI. The system is referred to as "Liquid MDI." The untreated elastomer has a softening point of 130°C for room temperature cured material (Figure 11) and 125°C for 71°C cured material (Figure 12). The traces for the hydrolyzed material were nearly the same for both cures, so the discussion below will be generalized for the two. After 24 hours, the samples had their softening points shifted to about 100°C and had severe surface blistering. At 48 hours, the softening points had lowered another 10°C, and the samples became very soluble, though they retained their resiliency well. GPC traces (Figure 22) revealed that the materials had degraded considerably, but only trace amounts of butanediol were found to be present. It is not known what the peak at 37.5 counts is. The samples were run on a low column set, which separates low molecular weight molecules better, and this peak vanished. After 96 hours (Figure 12), the samples were still resilient and not sticky, but their softening points were around 80°C. Infrared traces (Figure 17) displayed no evidence of free methylene dianiline (MDA) or butanediol; therefore, the molecule had not been completely ruptured but had only broken into smaller fragments of the same basic backbone.

Again it appears as though the oxygen to carbonyl bond has prevented complete hydrolysis. Recent evidence suggests that the cycloadduct will break down at elevated temperatures into a carbodiimide and MDI. Elevated temperature curing will cause this as will steam hydrolysis. The result is a linear elastomer with greater solubility and a reduction of physical properties. Very little is known of this mechanism or its effects at this time, but future work should clarify the situation. Apparently it is this mechanism which allowed the polymer to become rather quickly soluble and yet retain much of its resiliency.

Adiprene LW-520/MDA

LW-520 is a polyoxybutylene glycol, but in this case it is capped with Hylene W, which is 4,4'-dicyclohexylmethane diisocyanate. It is the only elastomer in this group which uses an aliphatic diisocyanate. LW-520 uses LD-2729 as an extender, and was cured

for 10 hours at 100°C. The untreated sample had a softening point of 170°C (Figure 13), which shifted to 135°C and became broader, after 24 hours of steam hydrolysis. Some surface blistering was also noted (Figure 2, specimen F). After 72 hours, the softening point had declined gradually to 60°C and remained broad. It was at this point that the elastomer became at least 50 percent soluble in THF. The infrared spectrum (Figure 18) contained basically the same peaks as in the untreated sample except that they were considerably sharpened, which is usually caused by a reduction in molecular weight. GPC traces (Figure 23) displayed a very broad distribution of molecular weights, including a good portion of high molecular weight material. At 96 hours (Figure 13) the elastomer melted while in the autoclave, though it re-solidified upon cooling. After 144 hours the softening point had declined to a very broad range centering at about 0°C. The sample still remained rubbery and non-sticky at room temperature. The GPC curve showed a considerable reduction in high molecular weight species, though some were still present, which accounted for its incomplete solubility. The peak at 37 counts was found to be MDA, thus the elastomer had broken entirely at the nitrogen to carbonyl bond. This would be predicted to happen, since that is where Adiprene L-100 cleaved.

Conap EN-7

EN-7 consists, unlike any of the other materials, of a polybutadiene glycol capped with TDI and extended with a mixture of 50 percent Isonol C-100, which is bis(2-hydroxypropyl) aniline, and 50 percent octylene glycol, which is 1,3-dihydroxy-2-ethylhexane. This system is cured with FeAcAc for 16 hours at 80°C. The untreated sample had a softening point of 200°C (Figure 14), which slowly declined to 74°C over a period of 216 hours of steam hydrolysis. The sample also exhibited a small depression at about 35°C, which appeared at 72 hours and gradually grew deeper throughout the 216 hours. From about 120 hours and above this depression would probably cause major property changes. With increasing exposure to steam the samples gradually darkened but never became soft or sticky. Their resistance to hydrolytic attack seemed excellent. None of the samples were even partially soluble and their infrared spectra (Figure 23) were identical to untreated sample spectra. It is evident that little cleavage occurred within the molecule under the test conditions.

ACCOMPLISHMENTS

The hydrolytic stability of four alternate materials to Adiprene, L-100/MOCA was measured by mechanical spectrometry, thermomechanical analysis (TMA), infrared spectrum analysis, solubility in tetrahydrofuran (THF), and gel permeation chromatography (GPC).

Conathane EN-7 was the most resistant to accelerated attack by steam hydrolysis showing little change in properties up to 50 hours exposure. Adiprene LW-520/MDA showed some effects due to 24 hours exposure. Adiprene L-100/MOCA, Adiprene L-315/polyol, and Uralite 3121S show poor stability to the accelerated hydrolysis environment.

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